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Indian Standard

SPECIFICATION FOR COPPER NAPHTHENATE

(*Second Revision*)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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SPECIFICATION FOR COPPER NAPHTHENATE

(*Second Revision*)

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(*Continued on page* 2)

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Indian Standard

SPECIFICATION FOR COPPER NAPHTHENATE

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 15 July 1987, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally issued in 1957. In the first revision, the limit of sulphonic acids and their derivatives were incorporated along with some changes in the methods of test and sampling procedure.

0.3 In this revision, behaviour on heating and requirement for copper have been amended. Alternate test methods for copper, chlorides and sulphates have been added and requirement for acid value deleted.

0.3.1 To correctly assess the quality of the material, it was felt to incorporate five new requirements, namely, iodine value, acid value, relative density, refractive index and product of relative density and refractive index of naphthenic acid extracted from the material and hence these parameters have been prescribed.

0.3.2 In the formulation of this standard, considerable assistance has been derived from Chief Inspectorate of Military Explosives (Kanpur), Ministry of Defence, and Projects & Development India Ltd, Sindri.

0.4 Copper naphthenate is extensively used in the rot-proofing of various textile fibres including ropes, sand bags, etc, and for fungicidal treatment of timbers. Copper content of the material is of great importance as its amount is related to its fungicidal and antirot properties. It is marketed as 8·0 percent copper concentrate and is often diluted before use.

0.5 Although the preservative value of the material was established as far back as in 1889, and minor amounts had been used for ship bottom paints and fish net preservatives, the real demand for copper naphthenate started during World War II when tremendous quantities of sand bags, camouflage nets, ropes and other military textiles and cellulose goods went into the battle zones of the pacific and similar areas infested with fungi and termites.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements, and methods of sampling and test for copper naphthenate.

2. REQUIREMENTS

2.1 Description — The material shall be the copper salt of cycloparaffin carboxylic acids extracted from mineral oils and shall be in the form of a hard solid or viscous liquid of deep green colour and shall not contain any solvent.

2.2 Behaviour on Heating — The material when heated to 100°C shall form a deep green viscous liquid, free from visible impurities and emitting a characteristic odour.

2.3 The material shall comply with the requirements prescribed in Table 1, when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of Table 1.

TABLE 1 REQUIREMENTS FOR COPPER NAPHTHENATE

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. OF APPENDIX A)
(1)	(2)	(3)	(4)
i)	Copper content (as Cu), percent by mass, *Min*	8·0	A-2
ii)	Matter insoluble in acetone, percent by mass, *Max*	5	A-3
iii)	Mineral acidity	To pass the test	A-4
iv)	Chlorides (as NaCl), percent by mass, *Max*	0·1	A-5
v)	Sulphates (as Na_2SO_4), percent by mass, *Max*	0·15	A-6
vi)	Sulphonic acids and their derivatives (as Na_2SO_4), percent by mass, *Max*	1·5*	A-7

*For special requirements of the Ministry of Defence, sulphonic acid and their derivatives shall be 0·5 percent by mass, *Max*.

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*Rules for rounding off numerical values (*revised*).

2.4 Requirements of Extracted Naphthenic Acid from Copper Naphthenate — The requirements of naphthenic acid extracted from copper naphthenate by the method given in **A-8** shall comply with the requirements prescribed in Table 2, when tested according to the methods prescribed in Appendix B. Reference to the relevant clauses of Appendix B is given in col 4 of Table 2.

TABLE 2 REQUIREMENTS FOR NAPHTHENIC ACID

SL NO.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. OF APPENDIX B)
(1)	(2)	(3)	(4)
i)	Acid value	170-240	B-1
ii)	Iodine value, *Max*	20	B-2
iii)	Relative density	0·94-1·0	B-3
iv)	Refractive index	1·47-1·52	B-4
v)	Product of (iii) and (iv)	1·38-1·47	B-5

3. PACKING AND MARKING

3.1 The material shall be packed in containers as agreed to between the purchaser and the supplier.

3.2 The container shall be securely closed and marked with the following information:

a) Name and net mass of the material;
b) Name of the manufacturer and/or his recognized trade-mark, if any;
c) Year of manufacture; and
d) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

3.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative samples of the material from a lot, number of tests to be performed and the method of finding out the criteria of conformity of the material to the requirements of this specification shall be as prescribed in Appendix C.

APPENDIX A

(*Clause* 2.3)

METHODS OF TEST FOR COPPER NAPHTHENATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070 - 1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF COPPER CONTENT

A-2.0 Two methods are prescribed for determining copper, namely, volumetric and spectrophotometric method. The latter method shall be the referee method.

A-2.1 Volumetric Method

A-2.1.0 *Outline of the Method* — Copper is determined with the addition of potassium iodide and titrating the liberated iodine against standard thiosulphate solution.

A-2.1.1 *Reagents*

A-2.1.1.1 *Dilute sulphuric acid* — approximately 2 N.

A-2.1.1.2 *Petroleum ether* — boiling range 40 to 60°C.

A-2.1.1.3 *Ammonium hydroxide* — approximately 18 N.

A-2.1.1.4 *Ammonium bifluoride solution* — saturated.

A-2.1.1.5 *Potassium iodide* — crystals.

A-2.1.1.6 *Standard sodium thiosulphate solution* — approximately 0·1 N, standardized against electrolytic copper.

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*Specification for water for general laboratory use (*second revision*).

A-2.1.1.7 *Starch indicator solution* — Triturate 5 g of starch and 0·01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into one litre of boiling water, boil for 3 minutes, allow the solution to cool and decant off the clear liquid.

A-2.1.1.8 *Ammonium thiocyanate* — solid.

A-2.1.1.9 *Benzene.*

A-2.1.2 *Procedure*

A-2.1.2.1 Weigh accurately 2 g of the material into a 250 ml beaker and dissolve in 100 ml of benzene. Heat and filter through filter paper No. 40. Wash the filter paper with hot benzene. Evaporate the filtrate on water bath till free from benzene. Add 100 ml of dilute sulphuric acid. Place the beaker on a sandbath, boil the contents and stir constantly for 30 minutes to prevent bumping. Boil for further 60 minutes (total time 90 minutes) and then cool. Transfer the liquid, which separates into two layers, to a separating funnel and then shake with 50 ml of petroleum ether. Draw off the lower aqueous layer into a 500 ml conical flask and wash the petroleum ether layer twice with 25 ml portions of water acidified with a few drops of dilute sulphuric acid each time.

A-2.1.2.2 Collect the washings in the conical flask (total volume about 150 ml), and neutralize by shaking with ammonium hydroxide until the solution gives a clear smell of ammonia. Add ammonium bifluoride solution till the smell of ammonia disappears and then 1 to 2 ml more. Cool the solution, add 2 g of potassium iodide and, after stoppering it, keep it for 2 to 3 minutes in a dark place. Titrate the liberated iodine with standard sodium thiosulphate solution, using starch solution as indicator, 2 g of ammonium thiocyanate being added to the mixture towards the end of the titration to sharpen the end point.

A-2.1.3 *Calculation*

$$\text{Copper content (as Cu), percent by mass} = \frac{6{\cdot}354\, VN}{M}$$

where

V = volume in ml of standard sodium thiosulphate solution,
N = normality of standard sodium thiosulphate solution, and
M = mass in g of the material taken for the test.

A-2.2 Spectrophotometric Method

A-2.2.0 *Outline of the Method* — The sample is dissolved in benzene and copper is extracted into aqueous phase with dilute hydrochloric acid. The absorbance owing to cupric ammonium complex is measured on spectrophotometer in red range.

A-2.2.1 *Reagents*

A-2.2.1.1 *Dilute nitric acid* – 1 : 1 (*v*/*v*).

A-2.2.1.2 *Concentrated hydrochloric acid* — *See* IS : 265 - 1976*.

A-2.2.1.3 *Benzene*

A-2.2.1.4 *Ammonium hydroxide*

A-2 2.1.5 *Standard copper solution* — Dissolve 2·000 g copper (99·9 percent pure) in 25 ml (1 : 1) nitric acid. Evaporate, add 10 ml concentrated hydrochloric acid. Again evaporate and add 100 ml concentrated hydrochloric acid. Make up the volume to exactly 1 000 ml.

1 ml = 0·002 g copper

A-2.2.2 *Preparation of Copper Calibration Graph* — From the standard copper solution, prepare the calibration solutions of the following concentrations by adding 20 ml of 20 percent hydrochloric acid and 20 ml of ammonium hydroxide, then making the volume up to 100 ml in each case:

a) Blank containing 20 ml of 20 percent hydrochloric acid, 20 ml of ammonium hydroxide and made up to 100 ml.
b) 15 ml of standard solution.
c) 20 ml of standard solution.
d) 25 ml of standard solution.

Measure the absorbance against blank at 620 nm in stoppered cuvettes in a spectrophotometer and plot the absorbance reading versus copper in g used in each of calibration solution.

A-2.2.3 *Procedure* — Weigh accurately about 0·5 g sample in a beaker. Dissolve in about 20 ml benzene and filter to a separating funnel washing beaker with small amounts of benzene. Extract copper with 2 portions of 10 ml of 20 percent hydrochloric acid. Wash benzene layer twice with 5 ml portions of 1 percent hydrochloric acid. Combine all extracts along with washings in a 100 ml volumetric flask. Add 20 ml ammonium hydroxide and dilute to the mark. Measure absorbance of the above sample solution at 620 nm in 10 mm cuvette in a similar way as above.

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*Specification for hydrochloric acid (*second revision*).

A-2.2.4 *Calculation*

$$\text{Copper, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M_1=mass in g of copper read from the graph, and
M=mass in g of the sample taken for the test.

A-3. DETERMINATION OF MATTER INSOLUBLE IN ACETONE

A-3.1 Procedure — Weigh accurately about 5 g of the material into a tared sintered glass crucible and place it in the Soxhlet apparatus in the position where the thimble is generally placed. Extract with sufficient quantity of acetone for 2 hours. Remove the sintered glass crucible from the Soxhlet apparatus and drive off acetone by suction as far as practicable. Then dry the residue in the sintered glass crucible to constant mass at 100 ±2°C.

A-3.2 Calculation

$$\text{Matter insoluble in acetone, percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1=mass in g of the dried residue, and
M =mass in g of the material taken for the test.

A-4. TEST FOR MINERAL ACIDITY

A-4.1 Prepared Sample Solution — Weigh accurately about 10 g of the material into a chemically resistant glass flask. Add 200 ml of water and boil for 1 hour under a reflux condenser. After refluxing, remove the condenser and continue boiling until the volume of the aqueous extract is less than 100 ml. Stopper the flask and allow it to cool to room temperature. Transfer the aqueous extract to 100-ml volumetric flask and make up the volume with water. Use this *prepared sample solution* for the tests under **A-4.3**, **A-5.2** and **A-6.2**.

A-4.2 Reagent

A-4.2.1 *Bromophenol Blue* — Warm 0·1 g of bromophenol blue with 3 ml of 0·05 N sodium hydroxide solution and 5 ml of rectified spirit (*see* IS : 323 - 1959*) until dissolved; dilute with ethyl alcohol (20 percent by volume) to make up to 250 ml.

A-4.3 Procedure — Transfer 25 ml of the *prepared sample solution* (*see* **A-4.1**) by means of a pipette and add a few drops of

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*Specification for rectified spirit (*revised*).

bromophenol blue indicator. The material shall be taken to have satisfied the requirement of the test if no yellow colour is produced.

A-5. DETERMINATION OF CHLORIDES

A-5.0 Outline of the Method — Two methods have been prescribed, namely, volumetric and turbidimetric, of which the former shall be the referee method.

A-5.1 Volumetric Method

A-5.1.1 *Reagents*

A-5.1.1.1 *Concentrated nitric acid* — *See* IS : 264 - 1976*.

A-5.1.1.2 *Standard silver nitrate solution*— approximately 0·1 N.

A-5.1.1.3 *Standard ammonium thiocyanate solution* — approximately 0·1 N.

A-5.1.1.4 *Ferric ammonium sulphate solution* — Dissolve 10 g of ferric ammonium sulphate in sufficient freshly boiled and cooled water to make 100 ml. Add 1 ml of concentrated nitric acid.

A-5.1.2 *Procedure* — Transfer 100 ml of the *prepared sample solution* (*see* **A-4.1**) into a conical flask. Add 2 ml of concentrated nitric acid and 10 ml of standard silver nitrate solution. Titrate the solution with standard ammonium thiocyanate solution using 2 to 3 drops of the ferric ammonium sulphate solution as indicator. Carry out a blank determination at the same time.

A-5.1.3 *Calculation*

$$\text{Chlorides (as NaCl), percent by mass} = \frac{58{\cdot}45\,(V_1 - V_2)\,N}{M}$$

where

V_1 = volume in ml of standard ammonium thiocyanate solution required for the blank titration,

V_2 = volume in ml of standard ammonium thiocyanate solution required for the test with the material,

N = normality of standard ammonium thiocyanate solution, and

M = mass in g of the material taken under **A-4.1**.

A-5.2 Turbidimetric Method

A-5.2.1 *Apparatus*

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*Specification for nitric acid (*second revision*).

A-5.2.1.1 *Nessler cylinders* — 50 ml capacity (*see* IS : 4161 - 1967*).

A-5.2.2 *Reagents*

A-5.2.2.1 *Concentrated nitric acid* — *See* IS : 264 - 1976†.

A-5.2.2.2 *Silver nitrate solution* — 5 percent (*m/v*).

A-5.2.2.3 *Standard chloride solution* — Dissolve 1·649 g of sodium chloride in water and make up the volume to 1 000 ml. Pipette out of 10 ml of the solution, dilute with water and make up the volume to 100 ml. One millilitre of this solution contains 0·1 mg of chlorides (as Cl).

A-5.2.2.4 *Hydrogen peroxide* — 35 percent (*m/m*).

A-5.2.3 *Procedure* — Dissolve 1·0 g of the material in water. Transfer to a Nessler cylinder and add 1 ml of concentrated nitric acid, 1 ml of silver nitrate solution, dilute it to the mark with water. Carry out a control test in the other Nessler cylinder using 10 ml of standard chloride solution of the material, and the same quantities of other reagents in the same total volume of the reaction mixture. Stir both the solutions with glass rods and compare the turbidities produced in the two cylinders after 5 minutes.

A-5.2.3.1 The limit shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-6. DETERMINATION OF SULPHATES

A-6.0 Two methods are prescribed for the determination of sulphates, namely, gravimetric and turbidimetric ; the gravimetric method shall be regarded as the referee method.

A-6.1 Gravimetric Method

A-6.1.1 *Reagents*

A-6.1.1.1 *Dilute hydrochloric acid* — approximately 5 N.

A-6.1.2.2 *Barium chloride solution* — 12 percent (*m/v*).

A-6.1.2 *Procedure* — Transfer 100 ml of the *prepared sample solution* (*see* **A-4.1**) into a beaker. Add 2 ml of dilute hydrochloric acid and 10 ml of hot barium chloride solution. Boil the solution for 15 minutes and then allow to stand for 4 hours. Filter through a tared Gooch or tared sintered glass crucible G. No. 4. Wash the precipitate thoroughly with hot water till free from chlorides and dry to constant mass at 105 to 110°C.

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*Specification for Nessler cylinders.

†Specification for nitric acid (*second revision*).

A-6.1.3 *Calculation*

$$\text{Sulphates (as } Na_2SO_4 \text{), percent by mass} = \frac{608{\cdot}6\,A}{M}$$

where

A = mass in g of barium sulphate precipitate, and
M = mass in g of the material taken under **A-4.1.**

A-6.2 Turbidimetric Method

A-6.2.1 *Apparatus*

A-6.2.1.1 *Nessler cylinder* — 50 ml capacity.

A-6.2.2 *Reagents*

A-6.2.2.1 *Dilute hydrochloric acid* — *See* **A-6.1.1.1.**

A-6.2.2.2 *Barium chloride solution* — *See* **A-6.1.1.2.**

A-6.2.2.3 *Standard sulphate solution* — Dissolve 1·48 g of ignited sodium sulphate (Na_2SO_4) in water and dilute to 1 000 ml. Take 10 ml of this solution and dilute to 1 000 ml. One ml of this solution contains 0·1 mg of sulphate (as SO_4).

A-6.2.3 *Procedure* — Dissolve 1 g of sample in water and make up to 100 ml in a volumetric flask. Pipette 10 ml of solution into a Nessler cylinder. Acidify with 1 ml of 10 percent hydrochloric acid followed by 2 ml of barium chloride solution, make up to the mark and stir well. Any turbidity produced should be not more than that of 0·15 mg of sulphate, when treated in a similar manner as aliquot.

A-7. DETERMINATION OF SULPHONIC ACIDS AND THEIR DERIVATIVES

A-7.1 Reagents

A-7.1.1 *Sodium Hydroxide* — solid (*see* IS : 376 - 1976*).

A-7.1.2 *Bromine Water* — saturated solution.

A-7.1.3 *Dilute Hydrochloric acid* — same as in **A-6.1.1.**

A-7.1.4 *Barium Chloride Solution* — same as in **A-6.1.2.**

A-7.2 Procedure — Weigh 2·0 g of the material in a nickel crucible. Add 3 g of sodium hydroxide and about 2 ml of water. Mix with a

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*Specification for sodium hydroxide, analytical reagent (*second revision*).

nickel spatula. Heat the mixture slowly over a burner and then ignite the contents. After the burning is complete, heat the contents strongly until most of the carbon disappears. Cool the crucible, dissolve the contents in about 25 ml of water and then filter the solution into a beaker. Treat the filtrate with a few drops of bromine water till a slight yellow colour is produced and then acidify carefully with dilute hydrochloric acid. Boil the solution and add 2 ml of barium chloride solution. Continue boiling the mixture for 15 minutes and then allow to stand for 4 hours. Filter through a tared Gooch or tared sintered glass crucible G. No. 4. Wash the precipitate thoroughly with hot water till free from chlorides and dry to constant mass at 105 to 110°C.

A-7.2.1 Carry out a blank test with all the reagents used in **A-7.2.**

A-7.3 Calculation

a) Total sulphates (as Na_2SO_4),

$$\text{percent by mass} = \frac{60{\cdot}68\ (A-B)}{M}$$

where

A = mass in g of barium sulphate obtained with the material,

B = mass in g of barium sulphate obtained in the blank, and

M = mass in g of the material taken for the test.

b) Sulphonic acids and their derivatives (as Na_2SO_4), percent by mass $= X - Y$

where

X = percentage of total sulphates as obtained in **A-7.3**(a), and

Y = percentage of sulphates as obtained in **A-6.**

A-8. PROCEDURE FOR SEPARATING NAPHTHENIC ACID FROM COPPER NAPHTHENATE

A-8.1 Procedure — Weigh about 100 g material, add 300 ml of dilute sulphuric acid (2 N). Boil for 90 minutes and cool. Transfer the liquid which separates into two layers to a separating funnel and shake twice with 50 ml of petroleum ether (**A-2.1.2**). Reject the aqueous layer. Wash the petroleum ether layer with 25 ml of water acidified with a few drops of dilute sulphuric acid. Distil off the petroleum ether collected in a flask as far as possible on a water bath.

A-8.1.1 Add to the flask, 100 ml of normal alcoholic KOH (**B-1.1.1**) and 100 ml of water and swirl the mixture until all the naphthenic acids are brought into solution. Transfer the solution to a 500 ml separating funnel. Add 100 ml of petroleum ether and swirl.

Carry out two further extractions of this aqueous layer with 50 ml portions of petroleum ether to remove solvents and unsaponifiable matter.

A-8.1.2 Remove most of the alcohol in the aqueous layer on a water bath. Transfer to a separating funnel and make up the volume to approximately 200 ml with water. Liberate the naphthenic acids by adding 25 ml of concentrated hydrochloric acid (**B-1.1.3**) and extract them by shaking with 100 ml of petroleum ether. Wash the petroleum ether layer with 10 percent sodium chloride solution until free from mineral acid and then with water to remove any sodium chloride carried over by petroleum ether.

A-8.1.3 Run the petroleum ether into a beaker and distil off most of the petroleum ether on a water bath. Dry at about 60°C to constant mass.

NOTE — Constant mass being taken as a loss in mass of not more than 100 mg after 15 minutes drying interval.

APPENDIX B

(*Clause* 2.4)

METHODS OF TEST FOR NAPHTHENIC ACID

B-1. DETERMINATION OF ACID VALUE

B-1.0 General — Acid value is the number of milligrams of potassium hydroxide required to neutralize one gram of naphthenic acid.

B-1.1 Reagents

B-1.1.1 *Alcoholic Potassium Hydroxide Solution* — approximately 1 N.

B-1.1.2 *Petroleum Ether*

B-1.1.3 *Concentrated Hydrochloric Acid* — *See* IS : 265-1977*.

B-1.1.4 *Sodium Chloride Solution* — approximately 10 percent.

B-1.1.5 *Standard Potassium Hydroxide Solution* — 0·2 N.

B-1.1.6 *Rectified Spirit* — *See* IS : 323 - 1959†, neutralized to phenolphthalein indicator.

B-1.1.7 *Phenolphthalein Indicator Solution* — Dissolve 0·5 g of phenolphthalein in 100 ml of rectified spirit, 50 percent by volume, that has been previously neutralized to the indicator.

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*Specification for hydrochloric acid (*second revision*).

†Specification for rectified spirit (*revised*).

B-1.2 Procedure

B-1.2.1 Weigh accurately about 0·5 g of the naphthenic acid as obtained from **A-8**. Dissolve the acid in 50 ml of rectified spirit and titrate the solution with standard potassium hydroxide solution using phenolphthalein as indicator.

B-1.3 Calculation

$$\text{Acid value} = \frac{56{\cdot}1\,VN}{M}$$

where

V=volume in ml of standard potassium hydroxide solution used for the test,

N=normality of standard potassium hydroxide solution, and

M=mass in g of the naphthenic acids obtained in the test.

B-2. DETERMINATION OF IODINE VALUE

B-2.0 Outline of the Method — The iodine value of a material is a measure of its unsaturation. It is determined by the absorption of iodine under conditions as specified and is expressed in grams of iodine absorbed per 100 g of the material. For materials without conjugated double bonds, Wijs method for determination of unsaturation (iodine value) is most commonly used.

B-2.1 Wijs Method

B-2.1.1 *Reagents*

B-2.1.1.1 *Potassium dichromate* — *See* IS : 250-1964*.

B-2.1.1.2 *Concentrated hydrochloric acid* — *See* IS : 265-1976†.

B-2.1.1.3 *Potassium iodine solution* — Prepare a fresh solution by dissolving 10 g of potassium iodide, free from potassium iodate, in 90 ml of water.

B-2.1.1.4 *Starch solution* — Triturate 5 g of soluble starch and 0·01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for three minutes. Allow to cool and decant off the supernatant clear liquid.

B-2.1.1 5 *Standard sodium thiosulphate solution* — approximately 0·1 N. Dissolve approximately 24·8 g of sodium thiosulphate crystals

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*Specification for potassium bichromate, technical and analytical reagent (*revised*).

†Specification for hydrochloric acid (*second revision*).

($Na_2S_2O_3 . 5H_2O$) in water which has been well boiled to free it from carbon dioxide and make up to 1 000 ml. Store the solution in a cool place in a dark-coloured stock bottle with a guard-tube filled with soda lime. After storing the solution for about two weeks, filter, if necessary, and standardize it as prescribed below:

> Weigh accurately about 5 g of finely ground potassium dichromate which has been previously dried to a constant mass at 105±2°C into a clean one-litre volumetric flask. Dissolve in water, make up to the mark; shake thoroughly and keep the solution in a cool dark place. For standardization of sodium thiosulphate, pipette 25 ml of this solution into a clean glass-stoppered 250-ml conical flask or bottle. Add 5 ml of concentrated hydrochloric acid and 15 ml of 10 percent potassium iodide solution. Allow to stand in the dark for 5 minutes and titrate the mixture with the solution of sodium thiosulphate, using starch solution as an internal indicator towards the end. The end point is taken when the blue colour changes to green. Calculate the normality (N) of the sodium thiosulphate solution as follows:

$$N = \frac{25\ M}{49{\cdot}03\ V}$$

where

M = mass in g of the potassium dichromate, and

V = volume in ml of sodium thiosulphate solution required the titration.

B-2.1.1.6 *Iodine crystals* — re-sublimed.

B-2.1.1.7 *Acetic acid* — glacial, 99 percent, having a melting point of 14·8°C and free from reducing impurities. Determine the melting point of the acetic acid and test it for reducing impurities as follows:

a) *Determination of melting point* — Take a 15-cm long test tube and fill it to about two-thirds with the acetic acid. Insert into the acid a thermometer through a cork stopper fitting the test tube. The amount of acid shall be at least double the quantity required to cover the bulb of the thermometer when the bottom of the latter is 12 mm from the bottom of the test tube. Suspend this tube within a larger test tube through a cork. Cool the acid by immersing the assembly in ice water until the temperature is 10°C, then withdraw the assembly from the ice water and stir the acid rather vigorously for a few moments, thus causing the super-cooled liquid to crystallize partially. Take thermometer readings every 15 seconds and take the temperature at which the reading remains constant for at least 2 minutes.

NOTE — An engraved stem thermometer calibrated between 10°C and 65°C and in 0·1°C intervals and with the 0°C point marked on the stem, is recommended. The thermometer shall have an auxiliary reservoir at the upper end, a length of about 6 mm and a diameter of about 6 mm.

b) *Test for reducing impurities (potassium permanganate test)* — Dilute 2 ml of the acetic acid with 10 ml of water, add 0·1 ml of 0·1 N potassium permanganate solution and maintain at 27±2°C. The material shall be taken as free from reducing impurities if the pink colour is not discharged at the end of two hours.

B-2.1.1.8 *Chlorine gas* — dry.

B-2.1.1.9 *Iodine trichloride* — (ICl_3).

B-2.1.1.10 *Iodine monochloride (ICl)* — 98 percent.

B-2.1.1.11 *Wijs iodine monochloride solution* — Prepare this solution by one of the following three methods and store in a glass-stoppered bottle in a cool place, protected from light:

a) Dissolve 13 g of iodine in one litre of acetic acid, using gentle heat, if necessary, and determine the strength by titration with standard sodium thiosulphate solution. Set aside 50 to 100 ml of the solution and introduce chlorine gas into the remainder until the characteristic colour change occurs and the halogen content is nearly doubled as ascertained again by titration. If the halogen content has been more than doubled, reduce it by adding the requisite quantity of the iodine-acetic acid solution. A slight excess of iodine does no harm, but avoid an excess of chlorine.

Example

If the titration of 20 ml of original iodine-acetic acid solution requires 22 ml of standard sodium thiosulphate, 20 ml of the finished Wijs solution should require between 43 and 44 ml (and not more than 44 ml) of the same sodium thiosulphate solution.

b) Dissolve 8 g of iodine trichloride in approximately 450 ml of acetic acid. Dissolve separately 9 g of iodine in 450 ml of acetic acid, using heat, if necessary. Add gradually the iodine solution to the iodine trichloride until the colour has changed to reddish-brown. Add 50 ml more of iodine solution and dilute the mixture with acetic acid till 10 ml of the mixture is equivalent to 20 ml of standard thiosulphate solution when the halogen content is estimated by titration in the presence of an excess of potassium iodide and water. Heat the solution to 100°C for 20 minutes and cool. Prevent access of water vapour in preparing the solution.

c) Dissolve 10 ml of iodine monochloride in about 1 800 ml of glacial acetic acid and shake vigorously. Using safety pipette, take 5 ml of this, add 10 ml of potassium iodide solution and titrate with 0·1 N standard sodium thiosulphate solution, using starch solution as indicator. Adjust the volume of the solution till it is approximately 0·2 N.

B-2.1.1.12 *Carbon tetrachloride or chloroform* — inert to Wijs solution.

B-2.1.2 *Procedure* — Weigh accurately, an appropriate quantity of the naphthenic acid as obtained from **A-8** between the limits indicated in col 2 and 3 of Table 3, into a clean dry 500-ml iodine flask or well ground glass-stoppered bottle to which 25 ml of carbon tetrachloride or chloroform have been added and agitate to dissolve the contents. The mass of the material shall be such that there is an excess of 50 to 60 percent of Wijs solution over that actually needed. Add 25 ml of the Wijs solution and replace the glass stopper after wetting with potassium iodide solution; swirl for intimate mixing, and allow to stand in the dark for one hour. Carry out a blank test simultaneously under similar experimental conditions. After standing, add 15 ml of potassium iodide solution and 100 ml of water, rinsing in the stopper also and titrate the liberated iodine with standard sodium thiosulphate solution, swirling the contents of the bottle continuously to avoid any local excess until the colour of the solution is straw yellow. Add one millilitre of the starch solution and continue the titration until the blue colour formed disappears after thorough shaking with the stopper on.

TABLE 3 MASS OF NAPHTHENIC ACID TO BE TAKEN FOR DETERMINATION OF IODINE VALUE

IODINE VALUE EXPECTED	MASS IN g OF MATERIAL		WEIGHING ACCURACY
	Maximum	Minimum	
(1)	(2)	(3)	(4)
Less than 3	10·0	10·0	±0·001
5	6·346 0	5·077 0	±0·000 5
10	3·173 0	2·538 4	±0·000 2
50	0·661 2	0·528 8	±0·000 2

B-3. DETERMINATION OF RELATIVE DENSITY

B-3.1 Apparatus

B-3.1.1 *Relative Density Bottle or Pyknometer* — With well-fitting ground glass joints. To calibrate, clean and dry the bottle or pyknometer thoroughly, weigh and then fill with recently boiled and cooled water at about 25°C after removing the cap of the side arm. Fill to overflowing by holding the bottle or pyknometer on its side in such a manner as to prevent the entrapment of air bubbles. Insert the stopper and immerse in a water bath at the desired test temperature ±0·2°C. Keep the entire bulb completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, replace the cap, cool to room temperature and weigh. Calculate the mass of water. This is a constant for the bottle or pyknometer, but should be checked periodically. A pyknometer of about 50-ml capacity and of either of the two shapes as shown in Fig. 1 is recommended.

B-3.1.2 *Water Bath* — maintained at 30·0 ± 0·2°C.

B-3.1.3 *Thermometer* — any covenient thermometer of a suitable range with 0·1° or 0·2°C subdivisions.

B-3.2 Procedure — Fill the bottle with the oil previously cooled to about 25°C, holding the bottle on its side in such a manner as to prevent the entrapment of air bubbles after removing the cap of the side arm. Insert the stopper, immerse in the water bath at 30·0 ± 0·2°C and hold for 30 minutes. Carefully wipe off any oil which has come through the capillary opening. Remove the bottle from the bath, clean and dry it thoroughly. Replace the cap of the side arm, cool to room temperature and weigh.

B-3.3 Calculation

$$\text{Relative density at } 30^\circ/30^\circ\text{C} = \frac{A - B}{C - B}$$

where

A = mass in g of the bottle with sample at 30°C,
B = mass in g of the bottle, and
C = mass in g of the bottle with water at 30°C.

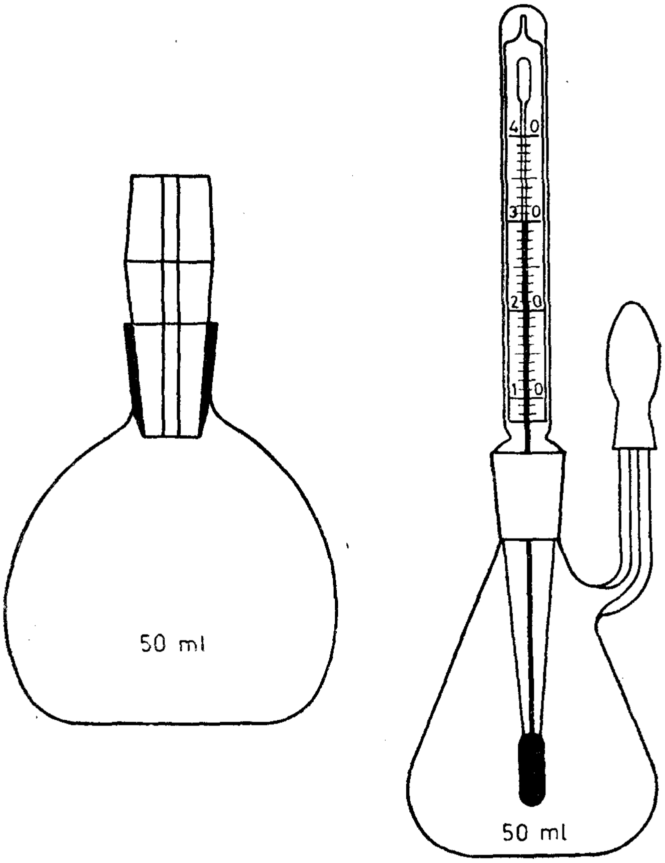

FIG. 1 PYKNOMETERS

B-3.3.1 *Temperature Corrections* — If the relative density is determined at any other temperature, then temperature corrections, as given under, shall be applied.

B-3.3.1.1 If the temperature of testing is higher than the specified temperature, add 0·000 65 to the value of the relative density obtained for each degree centigrade of the difference between the two temperatures.

B-3.3.1.2 If the temperature of testing is lower than the specified temperature, subtract 0·000 65 for each degree centigrade of the difference between the two temperatures.

B-4. REFRACTIVE INDEX

B-4.0 Outline of the Method — Refractive index is determined with an Abbe refractometer.

B-4.1 Apparatus

B-4.1.1 *Refractometer* — Abbe refractometer, the temperature of which shall be controlled to within ± 0·1°C and for this purpose, it shall be provided with a thermostatically controlled water bath and a motor-driven pump to circulate water through the instrument. The instrument shall be standardized, following the manufacturer's instructions, with a liquid of known purity and refractive index or with a glass prism of known refractive index. Water, which has a refractive index of 1·333 0 at 20·0°C, is a satisfactory liquid for standardization.

B-4.1.2 *Light Source* — If the refractometer is equipped with a compensator, a tungsten lamp or a daylight bulb shall be used. Otherwise, a monochromatic light, such as an electric sodium vapour lamp shall be used.

B-4.2 Procedure — Filter the material through a filter paper to remove any impurities and the last traces of moisture. Make sure that the sample is completely dry. Adjust the temperature of the refractometer to 40·0 ± 0·1°C or to any other desired temperature. Ensure that the prisms are clean and completely dry, and then place a few drops of the material on the lower prism. Close the prisms, tighten firmly with the screw-head and allow to stand for one or two minutes. Adjust the instrument and light to obtain the most distinct reading possible and determine the refractive index.

B-4.3 Temperature Corrections — Approximate corrections shall be made using the following equation:

$$R = R_1 + K(T_1 - T)$$

where

R = reading of the refractometer reduced to the specified temperature, T°C;

R_1 = reading at T_1°C;

T_1 = temperature at which the reading R_1 is taken;

T = specified temperature (generally 40·0°C); and

constant, K = 0·000 385.

B-5. PRODUCT OF RELATIVE DENSITY AND REFRACTIVE INDEX

B-5.1 Calculation

Product of relative density and refractive index = $A \times B$

where

A = relative density as determined in **B-3.3**, and

B = refractive index as determined in **B-4.2.**

APPENDIX C

(*Clause* 4.1)

SAMPLING OF COPPER NAPHTHENATE

C-1. GENERAL REQUIREMENS OF SAMPLING

C-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

C-1.1 Samples shall not be taken at a place exposed to the adverse effects of weather.

C-1.2 The sampling instruments and sample containers shall be clean and dry.

C-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

C-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers, on which the material has no action.

C-1.6 The sample container shall be of such a size that they are almost completely filled by the sample.

C-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the lot and batch numbers.

C-2. SCALE OF SAMPLING

C-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

C-2.1.1 Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of the specification.

C-2.2 The number (n) of containers to be selected at random from the lot shall depend on the size of the lot and shall be in accordance with Table 4.

TABLE 4 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE N (1)	NUMBER OF CONTAINERS TO BE SELECTED n (2)
Up to 25	2
26 to 50	3
51 to 100	4
101 and above	5

C-2.3 These containers shall be chosen at random from the lot. For random selection procedures, guidance can be had from IS : 4905 - 1968*. In case this standard is not readily available, the following procedure shall be adopted:

> Arrange the containers in the lot in a systematic order and starting from any one count them as 1, 2, 3, ..., etc., up to r and so on where r is the integral part of $\frac{N}{n}$ (N and n being the lot size and sample size respectively). Every rth container thus counted shall be withdrawn to constitute the sample.

C-3. TEST SAMPLES AND REFEREE SAMPLES

C-3.1 From each of the containers selected according to **C-2.2**, draw with an appropriate sampling instrument a representative portion of the material sufficient for carrying out the triplicate tests for all the requirements given in this specification.

C-3.2 Out of these portions, a small but approximately equal quantity of material shall be taken and mixed thoroughly to constitute a composite sample. The quantity of material in the composite sample shall be sufficient for triplicate tests required to be made on the composite sample (*see* **C-4.2**). The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample. Each part of the composite sample shall be transferred to separate bottles, sealed and labelled with full identification particulars (*see* **C-1.7**).

C-3.3 From the remaining portion of the material for each selected container, the quantity of material sufficient for triplicate tests required to be made on the individual samples shall be taken (*see* **C-4.1**). This shall be divided into three equal parts and transferred to separate bottles, sealed and labelled with full identification particulars of the samples (*see* **C-1.7**). The material in each such bottle shall constitute an individual sample. One set of bottles containing individual samples from all the selected containers shall be marked for the purchaser, another for the supplier and the third for the referee.

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*Methods for random sampling.

C-3.4 Referee Samples — The referee samples consisting of a composite sample (*see* **C-3.2**) and a set of individual samples (*see* **C-3.3**) shall bear the seals of both the purchaser and the supplier, and shall be kept at the place mutually agreed between them. These samples shall be used in case of a dispute between the two.

C-4. NUMBER OF TESTS

C-4.1 Tests for copper content of the material (*see* Table 1) and acid value as well as iodine value of naphthenic acid extracted from the material (*see* Table 2) shall be conducted on each of the sets of test samples representing individual samples.

C-4.2 Tests for the remaining requirements of naphthenic acid extracted from the material given in Table 2, that is, relative density, refractive index and product of relative density and reflective index, and for remaining requirements of the material given in Table 1, that is, matter insoluble in acetone, mineral acidity, chlorides, sulphates and sulphonic acid and their derivatives, shall be conducted on the composite sample.

C-5. CRITERIA FOR CONFORMITY

C-5.1 The test results for each of the characteristics tested on individual samples, namely, copper content of the material and acid value as well as iodine value of naphthenic acid extracted from the material shall be recorded.

C-5.1.1 Whenever the number of individual samples tested from a lot is two, both the test results shall meet the corresponding specification requirements for each characteristic, if the lot is to be considered as conforming to the requirements for these characteristics.

C-5.1.2 Whenever the number of individual samples tested from a lot is more than two, the average ($\bar{X}$) and range (R) of the test results shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of the test results}}{\text{Number of the test results}}$$

Range (R)=Difference between the maximum and the minimum values of the test results.

C-5.1.2.1 The lot shall be considered to have satisfied the requirements for these characteristics if the appropriate expression as shown in col 6 of Table 5 is satisfied for each of these characteristics.

C-5.2 For remaining requirements of the material as well as naphthenic acid extracted from the material (*see* **C-4.2**), all the test results on the composite sample shall meet corresponding requirements as specified in Tables 1 and 2.

C-5.3 The lot shall be declared as conforming to the requirements if **C-5.1** and **C-5.2** are satisfied.

TABLE 5 TEST RESULTS AND CRITERIA FOR CONFORMITY

(*Clause* C-5.1.2.1)

SL NO.	CHARACTERISTIC	TEST RESULTS 1, 2, 3......	AVERAGE	RATE	CRITERIA FOR CONFORMITY
(1)	(2)	(3)	(4)	(5)	(6)
i)	Copper content (as Cu), percent by mass, *Min*	1, 2, 3......	$\bar{X}_1$	R_1	$\bar{X}_1 - 0{\cdot}6R_1 \geqslant 8{\cdot}0$
ii)	Acid value	1, 2, 3......	$\bar{X}_2$	R_2	$\bar{X}_2 - 0{\cdot}6R_2 \geqslant 170$ $\bar{X}_2 + 0{\cdot}6R_2 \leqslant 240$
iii)	Iodine value, *Max*	1, 2, 3......	$\bar{X}_3$	R_3	$\bar{X}_3 + 0{\cdot}6R_3 \leqslant 20$

(*Continued from page* 2)

Members	*Representing*
Shri R. R. Ray	Waldies Ltd, Calcutta
Shri S. K. Barat (*Alternate*)	
Dr H. C. Shah	3 K Chemical Co, Thane
Shrimati K. H. Shah (*Alternate*)	
Shri S. R. Singh	Development Commissioner, Small Scale Industries, New Delhi
Shri J. C. Pasrija (*Alternate*)	
Shri S. S. Singhania	Forum of Small Scale Entrepreneurs, Calcutta
Shri B. L. Kanodia (*Alternate*)	
Dr J. K. Sinha	Central Mining Research Station (CSIR), Dhanbad
Shri V. B. Soni	Electrolytic Manganese Co Division of Union Carbide India Ltd, Thane
Shri D. Ghosh (*Alternate*)	
Dr M. S. Vaidya	Dharamsi Morarji Chemical Co Ltd, Bombay
Shri R. S. Vyas (*Alternate*)	